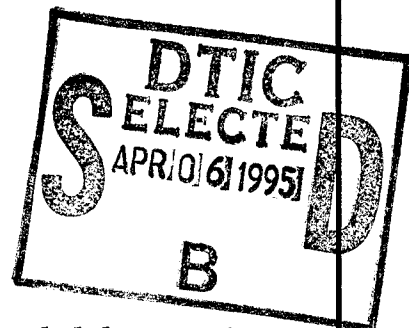


AOARD REPORT

Nippon Steel Research Laboratories, Futtsu, 28 Feb 94, and
Advanced Materials and Technology Research Laboratories,
Kawasaki, 15 Jun 94

June 15, 1994
P. McQuay
AOARD



Visits were paid to two of Nippon's Steels premier research laboratories: Steel Research and Advance Materials and Technology Research. The main focus of the visits were titanium, titanium-aluminide, and carbon-based materials development at Nippon Steel, all areas in which Nippon Steel is trying to develop new products and markets. In particular, Nippon Steel has been somewhat successful in marketing Ti as a structural and functional material in new non-aerospace markets such as construction and consumer goods. They have also placed emphasis on the automotive markets, but so far have met only limited success. Their penetration into the high performance aerospace Ti alloys markets has been very limited. Research and development efforts in new self-lubricating materials and functionally gradient materials technologies are also reviewed.

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AOARD Trip Report

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SUBJECT: Trip Report - Nippon Steel Research Laboratories, Futtsu, 28 Feb 94, and
 Advanced Materials and Technology Research Laboratories, Kawasaki, 15 Jun 94

1. ABSTRACT

Visits were paid to two of Nippon's Steels premier research laboratories: Steel Research and Advance Materials and Technology Research. The main focus of the visits were titanium, titanium-aluminide, and carbon-based materials development at Nippon Steel, all areas in which Nippon Steel is trying to develop new products and markets. In particular, Nippon Steel has been somewhat successful in marketing Ti as a structural and functional material in new non-aerospace markets such as construction and consumer goods. They have also placed emphasis on the automotive markets, but so far have met only limited success. Their penetration into the high performance aerospace Ti alloys markets have been very limited. Research and development efforts in new self-lubricating materials and functionally gradient materials technologies are also reviewed.

2. OVERVIEW AND BACKGROUND

Nippon Steel is the largest steel producer in the world. In terms of total crude steel production, Nippon Steel was ranked number one in the world in CY92, followed by Usinor-Sacilor of France, and POSCO of Korea, although in terms of total sales, Nippon Steel falls to number 4 behind Thyssen A.G. of Germany, USX of the USA, and Usinor-Sacilor of France (CY91).

Approximately 84% of Nippon Steels sales are from steel products, roughly 14% from engineering and construction, and the remaining 2% from chemical, nonferrous metal and ceramic products and electronics and information products (CY92). With the increasing competition by the aggressive Korean and Taiwanese steel mills, and a resurgent US steel industry aided by a weak dollar, Nippon Steel is aggressively attempting to further diversify into other advanced materials, chemicals, and electronic/communication industries. Their corporate business plan several years ago was to hold their steel business at a nearly constant level while dramatically increasing their sales of these other alternative products, in order to reduce the percentage of total sales of steels to 64%. Judging from the CY92 numbers, they have a long way to go.

The Japanese steelmakers have relied on technology and efficiency to remain profitable in spite of stiff competition. An example of this is Nippon's steels high continuous casting ratio, which is the tonnage of steel produced via continuous casting, divided by the total tonnage of steel produced. This ratio gives an indication of automation and efficiency, as continuous casting and other automation dramatically decreases the cost of steel. For Japan

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the ratio is 96.4%, and 99.1% for Nippon Steel. By comparison, the ratio for the USA steel industry is 68%.

It is interesting to observe the layout of a modern Japanese steel making works, where everything is designed for efficiency. Nearly all of the modern works are built on the Pacific coast of Japan, to facilitate the import of raw materials and export of finished products. The works are generally located on man-made islands which are specifically designed for the plants. The different process steps are highly integrated and nearly continuous, with the output from one process leading directly into the input to the next, with very little handling or transportation required. Most of the processes are heat and energy intensive, so this energy is conserved and utilized often by the next process in the process stream. Cargo ships berth at one end of the island in order to offload coal, iron ore, and other raw materials, and other ships are waiting on the other end to pick up the finished products. This integration has reduced the time it takes to reduce the ore, to completing the mill product from literally weeks or months to several days.

It is unclear, however, that any amount of efficiency and productivity can overcome the ever strengthening Japanese yen, which prices Japanese exports out of markets, and makes the increase of importation of foreign steel to Japan inevitable (which historically has been almost nonexistent). The combination of a weakening export market, and the loss of control of pricing and distribution in their domestic market, may make for very tough times for the Japanese steel industry. An analogy could obviously be made to many other sectors of the Japanese economy.

The total number of Nippon Steel employees for CY92 was approximately 50,000, down from approximately 80,000 in 1970. At the same time there has been a dramatic increase in the number of college graduates with technical degrees hired by Nippon Steel - another indication of a move toward less mature, high tech industries.

The Technical Development Bureau is responsible for research and development at Nippon Steel. It is currently divided into six laboratories or centers: Steel Research Laboratories; Process Technology Research Laboratories; Advanced Materials and Technology Research Laboratories; Electronics Research Laboratories; Plant Engineering and Technology Center; and Works R&D Laboratories. The remainder of this report will outline my visits to the Steel Research Laboratories and the Advanced Materials and Technology Research Laboratories.

3. NIPPON STEEL RESEARCH LABORATORIES, FUTTSU, 28 FEB 94

This visit was arranged by Mr. Yasushi Murakami, as a follow up visit following the Titanium Assessment Activity carried out in Dec 93 (see AOARD TR-94-15). My primary host at the laboratory was:

Mr. Mitsuo Ishii
Sr. Research Metallurgist
Nippon Steel
Stainless Steel and Titanium
20-1 Shintomi
Futtsu, Chiba-ken
Japan 299-12
Tel: +81 (439) 80-2288; Fax: +81 (439) 80-2745

Unfortunately, Mr. Ishii was slightly ill, although he was present for part of the visit. My other primary host was:

Dr. Hideki Fujii
 Sr. Researcher
 Nippon Steel
 Stainless Steel and Titanium
 20-1 Shintomi
 Futtsu, Chiba-ken
 Japan 299-12
 Tel: +81 (439) 80-2278; Fax: +81 (439) 80-2745

The Futtsu Research and Engineering Center is a new facility, only completed several years ago. Futtsu is in south Chiba Prefecture, on the peninsula across Tokyo bay, approximately an hour train ride from Tokyo. In addition to the Steel Research Laboratories, the Process Technology Research Laboratories and the Plant Engineering and Technology Center were also relocated at the Futtsu facility. In spite of the very nice grounds and impressive new facilities, nearly everyone complained about being so far from Tokyo.

In addition to steel research, the Steel Research Laboratories include the following Labs: Plate and Engineering Metallurgy ; Sheet and Coil; Surface Treatment; Electromagnetic Materials; Stainless Steel and Titanium; Bar, Shape and Wire Rod; Pipe and Tube; and Joining. I met with two groups of researchers from the Stainless Steel and Titanium, and the Joining Labs.

Nearly all of the R&D for conventional Ti and Ti alloys is conducted at Futtsu. As is pointed out in TR-94-15, there are some fundamental differences between the Ti industries in the US and Japan. In particular, the US Ti industry relies heavily upon the aerospace market and emphasizes Ti Alloy, whereas the Japanese Ti industry is only a minor player in the aerospace industry, and more than 85% of it's Ti products are commercially pure Ti (CP Ti).

The Japanese have been very aggressive in promoting non-aerospace uses of Ti, beyond the traditional chemical and oil and gas industries - the consumer products and the construction materials industry. Examples of innovative Ti consumer products marketed by Nippon Steel include jewelry, wine bottles and whiskey flasks, travel clothes iron, shears and scissors, knives, wrenches and other tools, and even a beverage thermos. Other Japanese Ti producers have succeeded in marketing watch cases and bands, mountaineering crampons, bike frames, and golf clubs. Examples of architectural or construction applications include ornamental and weather resistant roofs, gables, and beams for shrines, temples and other buildings, as well as various other external structures on high rises. Clearly, other than the novelty and the beauty of anodized Ti, one of the main selling points for the relatively expensive Ti, is the life-cycle cost benefits of Ti.

One of the more interesting construction applications for Ti are the Ti clad steel piers for the Tokyo Bay Highway Bridge which is currently under construction. The majority of the steel structure underwater is protected via coatings and sacrificial anodes, but the steel in the splash zone above and below the water level are protected by cladding with Ti (approximately 6 feet in height). The Ti and Steel plates are cold rolled together from plate, with the aid of a Cu insert sheet between them. Below is an abstract from a technical paper describing the process A. Yamamoto et al, Materials and Metals, v. 39,'92, pg. 62):

"A technique for hot rolling of the titanium clad steel sheet in atmospheric conditions has been developed. The titanium and steel sheets are assembled in the atmosphere through the copper sheet as insert metal between them. When such clad steel assembly is heated to high temperature, intermetallic compounds of titanium and copper are formed and melted in the interface. The air bubbles and oxides generated in the interface, are squeezed out from the interface with the aid of the melted intermetallic compound and compressive rolling pressure during hot rolling which results in the metallurgical bonding of clean metal surfaces of titanium and copper. On the other hand, copper sheet and steel can be easily bonded because of unstable oxides of both metals. In this technique, the most suitable heating temperature and time for the better bonding properties are 850 to 900°C for 300 to 1800 seconds. Titanium clad steel sheet manufactured using this technique shows approximately 180 MPa of shear strength. Such titanium clad steel is thought to have satisfactory performance with practical use, although its bonding strength is relatively lower than those of conventional titanium clad steel manufactured in vacuum condition."

The final gauge of the Ti clad steel sheet for the bridge application is 5 mm, where the thickness of the Ti face sheet and the steel plate is 1 mm and 4 mm, respectively. Nippon Steel is also pursuing other marine applications for its Ti products.

Like the US industry, the Japanese continue to aggressively pursue automotive applications, the veritable holy grail of the Ti industry. Just several widely used small parts of Ti such as automotive exhaust valves or coil springs would provide a relatively huge market for the Ti industry. Several Nippon Steel publications describing these marketing opportunities and efforts are available upon request:

"New Development of Titanium Application as an Ecological Metal - Non-Aerospace Application / Environment and Development"

"Outline of Anti-Wear Treatment for Titanium Alloy Intake Valves"

"Trends in Titanium Use by Japan's Automotive Industry"

"Applications of Titanium for Automotive Use"

Work on functionally graded materials (FGM) was being carried out by Nobuyuki Shimoda. Most of the research was carried out as part of "Research on the Basic Technology for the Development of Functionally Gradient Materials for Relaxation of Thermal Stress," by the Science and Technology Agency. The process for making the FGM is low-pressure plasma spray (LPPS), nominally on ceramic-metal systems. The ceramic was a $\text{ZrO}_2\text{-8wt\%Y}_2\text{O}_3$, and the metal was a Ni-20wt%Cr alloy.

Thermal loading tests were conducted of specimens by the National Aerospace Laboratory and Nippon Steel. In addition to a laser irradiation thermal shock test on small specimens, test specimens of approximately 100 mm x 50 mm were tested in a high-temperature gas flow test. The specimens were not found to have been degraded even after being subjected to very severe thermal gradients and thermal cycles (Kitaguchi et al, Nippon Steel Technical Report No. 57, Apr 93).

4. ADVANCED MATERIALS AND TECHNOLOGY RESEARCH LABORATORIES, KAWASAKI, 15 JUN 94.

The corporate basic or fundamental materials research center for Nippon Steel is the Advanced Materials and Technology Research Laboratory (AMTRL), in Kawasaki. AMTRL is staffed by 223 researchers, 94 technicians and 32 general staff (as of Jun 94). There are currently six research laboratories, each with the following emphases:

Chemicals (General Manager: Mr. Y. Nagashima): Specialty chemicals; Specialty polymers; Polymer and polymer alloy; Carbon fiber; Composites; Carbon and graphite; Physical chemistry.

Polymer Processing Development Center (GM: Dr. T. Herai): Polycarbonate sheet extrusion; Injection molding of structural components; Multilayer blow molding; Analysis by CAE.

Ceramic and Metals (GM: Dr. N. Okumura): Magnetic materials and devices; Metal and oxide superconductors; Structural ceramics; Ceramic-metal interface controlling technique; Integrated circuitry metals; Non-ferrous alloys.

Materials Characterization (GM: Dr. H. Kobayashi): Elemental and phase analysis; Surface analysis; Organic molecular analysis; On-line analysis; Electron microscopy and X-ray analysis; Materials science through characterization.

Future and Frontier Field (GM: Dr. M. Sakashita): Computational science; Surface-interface technology; Thin film technology; Innovative materials processing; Advanced materials technology.

Life Science Research Center (GM: Dr. I. Uno): Anti-tumor drugs; Cardiovascular agents.

This visit was also arranged by Mr. Yasushi Murakami, along with:

Dr. Keizo Hashimoto
Senior Researcher
Nippon Steel
Future and Frontier Field
Advanced Materials and Technology Research Laboratory
Technical Development Bureau
1618 Ida, Nakahara-ku
Tel: 044-777-4111 x3357; Fax: 044-752-6352

I was accompanied on this visit by Dr. Shiro Fujishiro, AOARD director. We were greeted and given an overview of AMRTL by Dr. Tomomi Murata, the Director of AMRTL, and Dr. Masao Sakashita, the GM of the Future and Frontier Field laboratory. We were also given a short briefing on four promising technologies currently under study: Development of bulk YBaCuO Superconductors; High Specific Strength TiAl Base Intermetallics; Hollow Fiber Membrane for Gas Separation; Low Temperature NbTi/Nb/Cu Multilayer Superconductors with High Formability and Workability. More information on these topics are available upon request.

Following the briefings, we were given a lab tour covering three areas: carbon fiber and carbon fiber reinforced plastics; metal-based self-lubricating composites; and intermetallic compounds.

The tour of the carbon-based materials lab was given by Dr. Kubomura. Nippon Steel is currently marketing a new pitch-based carbon fiber with high modulus. Many steel companies in Japan are trying to get into the pitch-based carbon fiber field, as pitch is a by product of steel making. They are also marketing several products using these new fibers. Several of the products were also of the sporting good variety: bike frames and special fishing rods or poles.

The tour of the self-lubricating materials lab was given by Dr. Nishida. Dr. Nishida's group has developed a new self-lubricating materials system based on tungsten disulfide crystals in an iron matrix. Two types of composites have evolved: the first is a high lubricity system, with a solid lubricant content of 70-80%, suitable for such applications as retainers of bearings for use under vacuum and high temperature environments (up to 350°C); the second is a high strength system with a lubricant content of 30-50%, suitable for gears and screws. A cold pressing/vacuum sintering process has been developed to reduce the production cost. They claim that the friction and wear characteristics are significantly better than the molydisulfide-moly-tantalum system, and that both types of composites are twice as strong as comparable conventional materials.

Work is under way now to extend the application ranges for high-strength composites, and to develop similar systems for use under atmospheric conditions at temperatures of up to 600°C. One promising system currently under study is a tungsten disulfide/boron nitride crystals in a stainless steel matrix.

The tour of the intermetallic compound lab was hosted by Dr. Hashimoto. Most of the funding for the laboratory has come through the MITI sponsored R&D Institute of Metals and Composites for Future Industries (RIMCOF), under the "High-Performance Materials for Severe Environments" project (see AOARD TR-93-005). This program has focused on primarily two intermetallic alloy systems, TiAl and Nb₃Al. The RIMCOF program is a cost sharing research consortium with many industrial and academic partners. The program is aimed at developing new high temperature materials for use in high mach aerospace vehicles and reusable spacecraft. The program is structured so that each of the members is given a small piece of the problem, and then cooperates with and shares information with the rest of the team, at least hypothetically. For example, Nippon Steel's assignment was to perform alloy development, and strip casting development for TiAl. Kobe Steel was assigned to develop an isothermal rolling mill and to perform rolling process development leading to the successful roll-forming of TiAl.

Nippon steel has made several significant contributions to research in TiAl. Several years ago they were successful in strip casting gamma TiAl, and subsequent development work has yielded sheet of good quality. A potential problem, however, is the direct rolling of as-cast TiAl sheet, which has a fairly coarse cast microstructure, and hence, very poor workability.

Dr. Hashimoto's group has also developed phase diagrams for several ternary alloy systems of importance: TiAl + Cr, V, Mo, and Nb. Based on the Ti-Al-Cr ternary work, Nippon steel was able to develop an alloy and microstructure through processing which achieved superplastic deformation of over 480% at 1130°C. At the processing and deformation temperature, a fine grained structure is retained by the grain pinning action of a grain boundary Cr-rich BCC phase. The actual superplastic forming work under the RIMCOF program, using the Nippon Steel alloy, is being done at Mitsubishi Heavy Industries (MHI) Nagoya Works, by Dr. Tsuzuka.

Nippon Steel has also achieved a refining process for TiAl which can reduce the typical interstitial oxygen content of the alloy to below 100 ppm by plasma arc melting. Typical values for cast TiAl made by induction skull melting or vacuum arc remelting are between 600 to 800 ppm. This is more remarkable considering they use standard Ti sponge and Al pellets, not high purity starting materials.

The work on Nb_3Al has been considerably less successful. The goals in the RIMCOF program are fairly aggressive in addressing the shortcomings of the material: poor oxidation resistance, poor room temperature ductility and formability or workability. The RIMCOF goals also include very aggressive high temperature strength goals. None of these technical problems have been completely overcome, even independently, and a clear development path to overcome these technical challenges has apparently not emerged.

5. SUMMARY AND COMMENTS

Nippon Steel is making concerted efforts to diversify into businesses beyond steelmaking: Electronics and Information, Chemicals, New Materials, and Services. In the New Materials field, they are concentrating on carbon and polymeric materials, aluminum, titanium, gamma titanium aluminide and ceramics as new structural materials. Certainly an advantage which many of the large Japanese steel companies possess in entering new areas of research and technology is their incredible wealth: financial, experiential, and technological.

The Japanese have been very aggressive in promoting non-aerospace uses of Ti, beyond the traditional chemical and oil and gas industries - consumer and construction products. Although particularly true of titanium, the same statement could probably be said for many other materials markets being pursued by Japanese corporations.

In the area of information technology and electronics, this strategy has evidently been successful so far. However, the timing of their attempted entrance into new markets of structural materials is unfortunate, as both the aerospace and automotive industries worldwide, with the possible exception of the US auto industry, are still depressed. However, they seem to have both the patience and the financial backing to make it in the long term.

Technical papers and summaries are available on most of the information discussed in this report.